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Patent

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| Application No. 10/625,060 | Confirmation No. 8260 |
| Applicant: Bryan B. Sauer | Filed: July 22, 2003 |
| Group Art Unit: 1794 | Examiner: Gray |
| Docket No. CL-1833 US NA | Customer No. 23906 |

## Declaration under 37 CFR §1.131

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

Sir:

Bryan B. Sauer does hereby declare as follows:

1. I am a citizen of the United States. I am a resident of Wilmington, Delaware, and I have been employed by E.I. du Pont de Nemours and Company in Wilmington, Delaware as a research chemist continuously since 1988.

2. I am an inventor of the subject matter described and claimed in the patent application identified above.

3. In the United States of America prior to July 17, 2001, I conceived, and completed, the production of fibers prepared from a

blend of a poly(ether ester), and an elastomeric propylene polymer, in which the propylene polymer was dispersed in a matrix of the poly(ether ester). The poly(ether ester) used was characterized by a low hard segment (26 wt% polybutylene terephthalate) content which is known to give high elasticity. The soft segment is based on a low glass transition dihydroxy polyether; poly(tetramethylene-co-2-methyltetramethylene ether). Weight average molecular weights of the poly(ether ester) were about 60,000 g/mol. Two types of propylene polymers were used: (a) a propylene stereoblock copolymer having 16% crystallinity, and (b) a propylene stereoblock copolymer having 32% crystallinity. These two polymers only contain propylene monomer but are sometimes referred to as copolymers because of the stereo-regular nature of the monomer arrangements.

4. The fibers were made by melt blending pellets of the constituent polymers at 230°C using the CSI melt mixer to provide extruded strands.. A ratio of 70 wt% of the poly(ether ester) and 30 wt% of the propylene polymer was used. These were then loaded in a separate step to a press spinner. The fibers produced had approximately 50 denier, or approximately 80 microns diameter. Once produced, the fibers were tested for either length after stretch, or for percent elongation.

5. The conduct and result of this work is described on the attached pages 12 and 13 of my data book E101131. On page 12, the chart at the top of the page shows the stretch and percent elongation test results for eight different runs in which fibers were prepared from a blend of 70 wt% poly(ether ester) and 30 wt% propylene polymer. In each run, the poly(ether ester) component is referred to as "8111". In runs 1~3 and 7~8 (counting from the left), the propylene polymer component is referred to as 101, which is a stereoblock copolymer having 16% crystallinity, and in runs 4~6, the propylene polymer component is referred to as 116, which is an stereoblock copolymer having 32% crystallinity. Fibers were melt-spun using a spinneret capillary with a length/diameter ratio (L/D) of

11 or 3. The charts on the bottom half of page 12 show a graphical presentation of the testing data.

6. On page 13, the production of additional blend fibers are described under the heading of "Example 6". The 30 wt% content for ELPP-D in the fibers refers to the propylene polymer component that is a stereoblock copolymer having 16% crystallinity, as discussed above, and the 30 wt% content for ELPP-B refers to the propylene polymer component that is a stereoblock copolymer having 32% crystallinity, also discussed above. The notations for all of the blend components are summarized in the column on the left side of page 13. Test results for the blends prepared as described on page 13 are shown at the bottom of that page in Table 4.

4. The actual work of the production and testing of the fibers as described above was done by me with some help by others on my research team. All of this work was performed and completed as described above in the U.S. prior to July 17, 2001.

5. The copies of data book pages E101131-12 and E101131-13 have been taken from my data book as issued to me by E.I. du Pont de Nemours and Company, my employer. I made the entries on these pages in the regular, ordinary course and conduct of my research work, and in the same manner as any other data entered in this or any other data book issued to and maintained by me. The copies that are attached are exact and unaltered photocopies of those respective pages except that all dates have been masked out. All of those dates are prior to July 17, 2001. Each page has been signed by me, and has been witnessed by Keisha M. Wilson.

6. I declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and declare further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. §1001, and that such willful false statements

may jeopardize the validity of this application or any patent issued thereon.

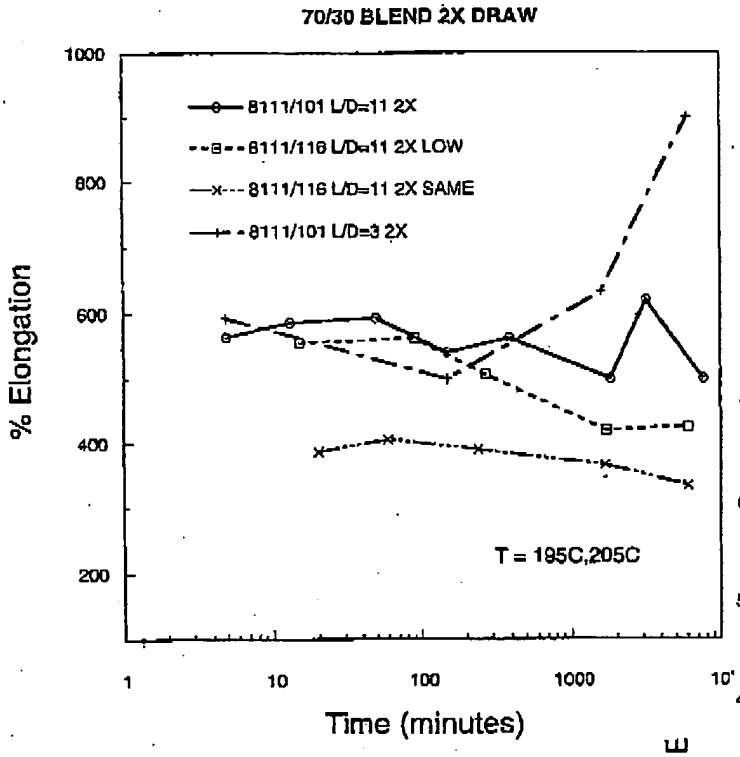
Bryan B. Sauer
Date signed: May 12, 2008

BOOK PAGE **E. I. du Pont de Nemours and Company**

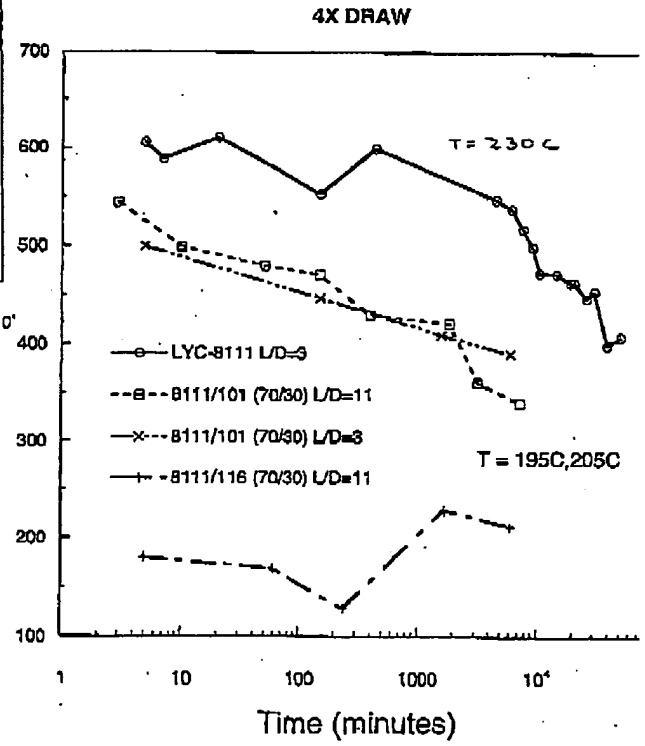
TITLE Excellent drops. of 1st DATE

**E 101131- 12** PURPOSE blend fibers, esp. wL -10# pp

| | 8111/101 (70/30) L/D = 11 1X | 8111/101 (70/30) L/D = 11 2X | 8111/101 (70/30) L/D = 11 4X | 8111/116 (70/30) L/D = 11 2X low dtu | 8111/116 (70/30) L/D = 11 2X same thru | 8111/116 (70/30) L/D = 11 4X | 8111/101 (70/30) L/D = 3 2X | 8111/101 (70/30) L/D = 3 4X |
|---|---|---|---|---|---|---|---|---|
| Length after 2X stretch 2cm to start | 3.3 | 2.05 | 2 | 2.1 | 2.2 | 2.1 | 2.05 | 2 |
| Length after 4X stretch 5 cycles 2 cm to start | 3.5 | 3.15 | 2.9 | 3.9 | 4.1 | broke | 3.05 | 3 |
| % ELONGATION -1 DAY | 1200 | 800 | 422 | 420 | 387 | 229 | 831 | 410 |




see pp. 9-10

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BOOK PAGE

TITLE Patent Examples For Citron DATE

PURPOSE Blend Fibers

E 101131- 13

Polyolefin Blend and Multi-component Fibers

This invention concerns fibers formed from two or more components. Both blends and core/sheath structures are of interest. One component is a low modulus thermoplastic polyolefin elastomer (ELPP). (We can give some relevant compositions or can define ELPP by its mechanical property limits, i.e. like recovery and modulus or elongation) Addition of less than 10% cross-linked polyolefin elastomer is covered in WO 97/45575. The other component is a segmented polymer including poly(ether ester), poly(ester ester), poly(ether amide), poly(ester amide), etc. Compositions include 0.5-80% ELPP, 99.5-20% segmented polymer. Invention provides processing improvements, lower cost, modified hydrophobicity, and modified water and dye absorption.

Bicomponent (side by side), nonwoven, and some non-textile melded blends are already covered.

ELPP-A: 13% crystalline polypropylene copolymer, WL-216
ELPP-B: 32% crystalline polypropylene copolymer, WL-116
PEE-C: 8111
ELPP-D: 16% crystalline polypropylene copolymer, WL-101

Example 1. Blends of ELPP with PEE exhibiting excellent recovery.

Blend films were made from 50% ELPP-A and 50% PEE-C. Materials were melt blended at 230C and pressed into films. Properties are indicated in Table 1 and show that % set of 50% ELPP/PEE is improved over 100% ELPP to values about the same as 100% PEE. A small decrease in elongation of the blend is also observed.

Example 2. Core/Sheath fibers with ELPP sheath and excellent properties.

As the PEE component is increased to about 40%, elastic fiber properties such as % E after aging begin to approach those of a 100% PEE and are far superior to 100% ELPP. Values of % E in Table 2 show that for a 67% ELPP-A/33% PEE-C fiber that properties are more stable with time than 100% ELPP-A, and that the % E after 60 days reaches 310%, similar to that for 100% PEE-C after 60 days, while %E for 100% ELPP-A after 60 days is 200%.

These fibers have hydrophobic surfaces, and processing of fabrics and other use properties will be affected by the mostly hydrophobic ELPP fraction.

Example 3. Core/Sheath fibers with high ELPP content.

Even at very high ELPP fractions of about 70%, fibers with elongations a factor of 1.5 better than 100% ELPP are obtained. (Table 2)

Example 4. ELPP-A Core/PEE-C Sheath.

Fibers were spun where sheath and core materials were reversed to give hydrophilic surfaces and higher melting components on the surface. Mechanical properties are consistent with those in Examples 2-3 for similar compositions.

Example 5.

Comparative example where lower elasticity and higher crystallinity ELPP was used in core/sheath fiber giving substantially lower elongations (Table 3) and elastic recovery.

Example 6. Blend fibers with excellent elongation, recovery, and processing

Blends were made by melt-mixing at 220C and fibers were spun with 4X drawing. Elastic properties in Table 4 for 30% ELPP-D/70% PEE-C show very high %E compared to 100% ELPP-D. Blends with harder ELPP-B show substantially reduced elongations and a high % set. Addition of ELPP components lowered processing temperatures considerably.

Table 1 Blend Films
(%E at break and % set after 5 cycles to 300%)

| | 100% ELPP-A | 50% ELPP-A | 100% PEE-C |
|---|---|---|---|
| % set | 110% | 70% | 75% |
| %E | 850% | 530% | 750% |

Table 2. ELPP-A Sheath/PEE-C Core
(4X Drawn Fibers)

| | 100% ELPP-A | 71% ELPP-A | 67% ELPP-A | 56% ELPP-A | 100% PEE-C |
|---|---|---|---|---|---|
| Fresh* | 330% | 315% | 400% | 440% | 580% |
| 30 days | 215% | 255% | 370% | 290% | 400% |
| 60 days | 200% | 255% | 360% | 285% | 380% |

* (< 5 min after spinning)

Table 3. %E versus time for 4X fibers including blends with higher modulus ELPP
(ELPP-B Sheath/PEE-C Core)

| | 100% ELPP-B | 50% ELPP-B | 100% PEE-C |
|---|---|---|---|
| Fresh | 150% | 210% | 580% |
| 5 day | 85% | 105% | 460% |
| 20 day | 85% | 100% | 400% |

Table 4. 4X Blend Fibers with PEE-C as one component
(%E at break, % set after 5 cycles to 300%, and % recovery after 100% elongation)

| | 100% ELPP-D | 50% ELPP-D | 30% ELPP-B | 100% PEE-C |
|---|---|---|---|---|
| % set | broke | 45% | broke | 50% |
| %E | 200% | 420% | 230% | 560% |
| % recovery | 93 % | 97% | 95% | 97% |

10[5]

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